

Training **Identification of Cocaine and Crack**

I. Introduction:

Cocaine salt and Crack (Cocaine base) samples will be analyzed by a microcrystalline test utilizing TLTA (di-p-toluoyl-l-tartaric acid) as a reagent. For cocaine samples, TLTA alone will produce crystallization. However, for crack samples, TLTA and Acetic Acid is used to produce crystallization. The purpose of the microcrystalline test is to determine the presence of the l-cocaine diastereoisomer. Further confirmation of cocaine is performed by GC/FID and GC/MS analysis.

II. Reagents:

- A.) TLTA (di-p-toluoyl-l-tartaric acid): 10 mg TLTA, 1 mL Ethanol, 1 mL glycerine in 8 mL distilled water.
- B.) Acetic Acid (HOAC)
- C.) 9:1 Methylene Chloride/Isopropynol or Ethanol.
- D.) Methanol (solvent rinse for GC)
- E.) 0.1 N HCL: add 8.3 mL concentrated HCL to sufficient water to make 1 L. (quantification).
- F.) Cocaine HCL in C₂₄H₅₀ (quantitative standard).
- G.) 10% K₂HPO₄: dissolve 10g K₂HPO₄ in sufficient water to make 100 mL. (quantification).
- H.) C₂₄H₅₀ in Methylene Chloride (quantitative internal standard).
- I.) Anhydrous Sodium Sulfate (Na₂SO₄).

III. Equipment:

- A.) Microscope and slides
- B.) Analytical balance
- C.) Weigh paper
- D.) Pipettes
- E.) 50 mL volumetric flask
- F.) 25 mL volumetric flask
- G.) Stoppered test tubes (2)
- H.) 2 mL autosampler vials with Teflon caps
- I.) GC/FID: HP 6890 or HP 5890 series
- J.) GC/MS: HP 5890/5972 or HP 6890/5973 series.

IV. Procedure:

- A.) Microcrystalline Test:

1. On a microscope slide add a small amount of sample.
2. Add one drop of TLTA reagent to sample.
3. Observe under microscope at 100-125X. Crystallization should occur within a few minutes if the l-cocaine diastereoisomer is present.
4. If the sample does not crystallize within a few minutes, add one drop of HOAC to the sample on the slide. Observe under microscope at same magnification. Crystallization should occur within a few minutes, indicating that the sample is the free-base form of cocaine (crack).

B.) Chromatography by GC/FID and GC/MS.

1. Add about 5 mg of sample to a labeled 2 mL autosampler vial.
2. Add 1-2 mL of Ethanol or 9:1 Methylene Chloride/Isopropynol to the vial and cap.
3. Place vial(s) on the GC/FID autosampler and run with the following sequence: Standard, Blank, Samples, Standard.
4. GC/FID conditions are as follows:
Method: EXP.M
Oven:
Initial Temp: 245°C
Initial Time: 0.00 min.
Rate: 10°/min.
Final Temp: 290°C
Run Time: 10 min.
Max. Temp: 325°C
Equilibration Time: 0.5 min.
Inlet:
Mode: split (35:1)
Initial Temp: 250°C
Pressure: 24.99 psi
Gas Type: Helium
Column:
Capillary: HP-1 30m x 320um
Initial Flow: 3.3 mL/min.
Detector:
Temp: 300°C
Hydrogen Flow: 30.0 mL/min.
Air Flow: 400 mL/min.
Makeup Gas: Helium
5. Obtain chromatographs. If cocaine is present, the instrument will detect a peak with a retention time

around 3.40 minutes and will generate a report with accompanying chromatograph.

6. Check concentration to determine if dilutions are needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates that the sample may have to be reinjected.
7. Place same sequence on the GC/MS autosampler and run.
8. GC/MS conditions are as follows:
Method: EXP.M
Oven:
Initial Temp: 230°C
Initial Time: 0.00 min.
Max. Temp: 325°C
Equilibration Time: 0.50 min.
Rate: 10°/min.
Final Temp: 280°C
Run Time: 10 min.
Inlet:
Mode: split (50:1)
Initial Temp: 250°C
Pressure: 31.65 psi
Gas Type: Helium
Column:
Capillary: HP-1MS 25m x 200um x 0.33um
Max. Temp: 300°C
Initial Flow: 1.0 mL/min.
9. If cocaine is present in sample, the instrument will detect a total ion peak at approximate retention time of 3.9 minutes and will generate a report along with accompanying chromatograph and spectra. The spectra will contain the identity of the peak, its ion abundance and the ratio of ions 152:150, confirming the presence or absence of cocaine and the l-cocaine diastereoisomer. (see graph, last page)

C.) Quantitative Procedure:

1. Extract sample by weighing out 100 mg of sample in a 50 mL volumetric flask and bring to volume with 0.1 N HCL.
2. Record exact weight and calculate sample amount by dividing the weight by the final volume.

3. Prepare cocaine standard by weighing out 25 mg of Cocaine HCL in a 25 mL volumetric flask and bringing to volume with C₂₄H₅₀ in Methylene chloride.
4. In first stoppered test tube, labeled standard, add 2 mL of standard solution (prepared in step#3), 2 mL of 0.1 N HCL, and 1 mL of K₂HPO₄.
5. In second stoppered test tube, labeled with sample number, add 2 mL of sample (prepared in step#1), 2 mL of 0.1 N HCL, and 1 mL of K₂HPO₄. Two layers will form in each tube.
6. In labeled autosampler vials, add enough sodium sulfate (Na₂SO₄) to cover the bottom of the vial.
7. From each of the test tubes, pipette the bottom layer into the appropriate vial and cap.
8. Run the quantitation on the GC, with the following sequence: Cocaine Standard (inj.#1), Cocaine Standard (inj.#2, Calibration), Cocaine Standard (inj.#3), Blank, Sample(s), Cocaine Standard (inj#4).
9. Check the standard to make sure recovery is at 100%. If not, rerun the standard (possibly at a higher injection amount).

V. Results:

- A.) Report a positive result for Cocaine when TLTA crystals match TLTA crystals for Cocaine standard, GC and GC/MS retention times and spectra match standard Cocaine.
- B.) For the microcrystalline test, record in logbook as TLTA positive, TLTA positive with HOAC, TLTA negative, or TLTA negative with HOAC.
- C.) Record results of the GC/MS in logbook. Then transfer the results to appropriate sample cards that came with the actual samples. Be sure to include date of analysis, results, the number of tests performed per sample, and initials.
- D.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.
- E.) Crack Cocaine requires addition of HOAC to the TLTA to form crystals. Crack results are recorded for survey purposes and are not reported on certificates.